

3. "Fluidextract Aconite No. 2, 910,036A. Biologically standardized." Assayed chemically—14.3 mg. per 10 cc.
4. "Tincture Aconite No. 1, 3,071,282. Standard-M. L. D. 0.0004 cc. per Gm. body weight." Assayed chemically—13.6 mg. per 100 cc.
5. "Tincture Aconite No. 1, 909,454A. Biologically standardized." Assayed chemically—14.0 mg. per 100 cc.
6. "Tincture Aconite 'E.' " Assayed chemically—11.0 mg. per 100 cc.
7. "Tincture Aconite for Experimental Purposes." Assayed chemically—14.1 mg. per 100 cc.

Of the three samples of Fluidextract of Aconite, all of them met the requirements which we calculated to be equivalent to the N. F. requirements, that is, they contained at least 14 mg. of aconitine per 10 cc.

Of the four samples of Tincture of Aconite, one (Sample No. 6) should be eliminated because it was not indicated to be of U. S. P. strength, and one (Sample No. 4) fell below the strength we calculated to be equivalent to that of the U. S. P., that is, 14.4 to 15.7 mg. per 100 cc.

The other two tinctures met the requirement calculated to be equivalent to the U. S. P. requirement.

This is further evidence that the chemical assay process for Aconite and its preparations as recommended by us should be substituted for the present U. S. P. process.

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### VOLATILE OIL FROM WESTERN YARROW.\*

BY R. L. MCMURRAY.<sup>1</sup>

Considerable discussion has occurred as to whether the yarrow of the western states is the same species as that which grows in the eastern states. In 1834 Thomas Nuttall (1) classified the western yarrow as *Achillea lanulosa* Nuttall. This work was made on material collected while with the N. B. Wyeth expedition in the region of the falls of the Columbia River east to the first navigable waters of the Missouri River. The Index Kewensis (2) lists this not as a new species, as did Nuttall, but the same plant as *Achillea Millefolium* Linné. C. V. Piper (3) made a third classification by combining the preceding and giving it the name *Achillea Millefolium lanulosa* (Nutt.) Piper. Since taxonomists disagree on this plant, or plants, it was considered of possible interest to determine the constants of the volatile oil and compare them with those reported for other volatile oils of *Achillea Millefolium* Linné.

The material was gathered from various local habitats in Whitman County, Washington, during the second week of July, 1935. The flower-heads were clipped off and collected into bags. The stems and leaves were rejected. All foreign materials were carefully excluded. Collecting was done during the entire day. The flower-heads amounted to about 76 pounds in the fresh condition. They were packed the same day into a small steam distillation apparatus (capacity about 20 pounds of material) and distilled with "high pressure" steam.

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Careful note was taken during the entire process of distillation to observe when the azulene might come over; the question arose some time ago as to whether this constituent distilled over at the beginning or toward the end of the distillation. No blue color was observed to come over at any time, even though the distillation was continued 3 hours after the oil was considered to be entirely dispelled from the plant. On the other hand, the eastern yarrow yields this blue color (due to azulene, a hydrocarbon) by distilling both the fresh and dried plants and from the petroleum ether extract (4) of the dried plant. Thus azulene is apparently absent from the western yarrow.

The 76 pounds of clipped fresh flower-heads yielded 108.3 cc. of colorless volatile oil. In addition the aqueous distillate produced 8.4 cc. of colorless volatile oil by cohobation. A total of 116.7 cc. of volatile oil was obtained, equivalent to 0.305 per cent of the clipped fresh flower-heads.

The following constants were obtained for this volatile oil (oil recovered by cohobation was excluded) of *Achillea Millefolium lanulosa* (Nutt.) Piper:

Specific gravity at 25° C.	0.8995
Specific rotation at 25° C.	-8.98
Refractive index at 25° C.	1.4657
Acid number	1.82
Saponification number	26.75
Ester number	24.93

The following constants were determined in 1931 by the author for Wisconsin volatile oil of *Achillea Millefolium* Linné:

Specific gravity at 25° C.	0.9066
Specific rotation at 25° C.	-14.11
Refractive index at 25° C.	1.4703

Roland Kremers (5) reported the following constants for a volatile oil from a Wisconsin crop of *Achillea Millefolium* Linné grown in 1919:

Specific gravity at 17° C.	0.913
Acid number	4.27
Saponification number	10.92
Ester number	5.65

Thus the volatile oil from the western yarrow differs from that of the eastern yarrow in color and in the chemical and physical constants. This difference may be due to soil, climate or to being a different species of *Achillea*. If soil or climate are the direct factors, growing the western yarrow in an eastern habitat for several generations should produce a blue oil but, on the other hand, if the western yarrow is truly a different species it is doubtful if change of climate or soil would have so much an effect on the volatile oil.

This work has indicated that laboratory control would be necessary for acceptance of products from a new geographical source, even though taxonomists may insist that the plant concerned is identical with that from other habitats

## REFERENCES.

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- (3) Piper, C. V., and Beattie, R. K., "Flora of Southeastern Washington," 262 (1914).
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## THE DETERMINATION OF SMALL QUANTITIES OF FLUORINE IN DICALCIUM PHOSPHATE.\*

BY S. E. HARRIS AND W. G. CHRISTIANSEN.<sup>1</sup>

## INTRODUCTION.

The physiological effect of fluorine has been the subject of numerous studies in recent years. It has been shown by experiments upon animals that fairly large doses have a definite retarding influence upon growth. This was established by the use of rock-phosphate, containing 3-4% fluorine, to supply a mineral supplement in the diet of farm animals. The animals receiving this supplement did not flourish as did animals receiving calcium-phosphorus supplements in other forms, and the undesirable effects were traced to the presence of fluorine.

In smaller quantities, fluorine is found to affect the teeth. McClure and Mitchell (1), in studies made upon rats, found that in spite of earlier evidence to the contrary (2) there was no difference between soluble and insoluble fluorides in this respect. DeEds and Thomas (3) in a study of the effect of various fluorides and fluosilicates upon the teeth of male white rats, reported that the solubility of the fluorine compound was not a factor in its toxic effect, and that amounts of 0.5-1.0 mg. of fluorine per day per Kg. body weight can produce definite signs of toxicity. Smith and Leverton (4) also concluded, from experiments upon rats, that the solubility of the fluoride bears no relation to the amount which will cause initial tooth damage. Smith (5, 6, 7) in a study of the effect of drinking water upon children during the formation of the permanent teeth found that mottled teeth were always associated with the use of water containing more than 0.9 parts per million of fluorine.

Dicalcium phosphate has been found to be a valuable dietary supplement in doses of 3-5 Gm. daily. The phosphoric acid from which it is manufactured is derived from fluorine-bearing phosphatic rock, and a method for the determination of fluorine in the dicalcium phosphate is necessary, since traces of fluorine may persist through the manufacturing operations, and remain as calcium fluoride.

Various methods of determining fluorine in relation to phosphatic materials have been studied by Reynolds (8) who recommends the procedure of Willard and Winter (9, 10). This is based upon the distillation of the sample with 60% perchloric acid, the fluorine being volatilized in the form of hydrofluosilicic acid. It is determined in the distillate by titration with thorium nitrate solution, using a sodium alizarin sulphonate-zirconium nitrate lake as indicator. The method is rapid and accurate and can be used without recourse to special apparatus. In a

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